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COUPLING AUTOMATED ELECTRON BACKSCATTER DIFFRACTION WITH TRANSMISSION ELECTRON AND ATOMIC FORCE MICROSCOPIES

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Grain boundary network engineering is an emerging field that encompasses the concept that modifications to conventional thermomechanical processing can result in improved properties through the disruption of the random grain boundary network. Various researchers have reported a correlation between the grain boundary character distribution (defined as the fractions of "special" and "random" grain boundaries) and dramatic improvements in properties such as corrosion and stress corrosion cracking, creep, etc.[1-3] While much early work in the field emphasized property improvements, the opportunity now exists to elucidate the underlying materials science of grain boundary network engineering. Recent investigations at LLNL have coupled automated electron backscatter diffraction (EBSD)[4] with transmission electron microscopy (TEM)[5] and atomic force microscopy (AFM)[6] to elucidate these fundamental mechanisms.

An example of the coupling of TEM and EBSD is given in Figures 1 – 3. The EBSD image in Figure 1 reveals "segmentation" of boundaries from special to random and random to special and low angle grain boundaries in some grains, but not others, resulting from the 15% compression of an Inconel 600 polycrystal. TEM characterization of a similarly deformed sample corroborates both these observations. The grain on the left in Figure 2 is in a soft deformation orientation and exhibits tangles of dislocations while dislocation-rich bands are observed in the grain on the right, which is in a hard deformation orientation. These dislocation-rich bands also interact with the boundary to locally alter the misorientation from near a $\Sigma 91_d$ to near a $\Sigma 37_c$. Such observations validate the initial observation of the segmentation of the boundary observed in the EBSD image of Figure 1. Figure 3 is an example of the effect of annealing twinning on the change in misorientation across a boundary (Fig. 3a) and the formation of a Σ3, Σ9, and Σ27 triple junction (Fig. 3b) as commonly observed in grain boundary network engineered materials. These fine-scale details are correlated to mesoscopic observations of a high fraction of three special boundaries forming a triple junction (Fig. 3c).

To evaluate the corrosion properties of individual boundaries in various Ni-base alloys, EBSD has been coupled with AFM. Figure 4a is an AFM topological image of an Alloy C-22 sample corroded in HCL for 8 hrs and scanned with EBSD. Corrosion is observed along random boundaries but not at the Σ3 boundary. It is interesting to note that the random boundary exhibits different corrosion behavior on either side of the twin boundary. Figure 4b is an AFM topological image (depth of corrosion in grey scale) of an electropolished Inconel 600 sample overlaid with a misorientation map indicating special boundaries in black and random boundaries in white. Triple junctions appear to be the preferred sites for the initiation of the earliest stages of localized attack.

This investigation provides evidence that grain boundary network engineering and the formation of annealing twins disrupt the connectivity of the random grain boundary network and is likely responsible for the experimentally observed improvement in properties. This work illustrates that coupling of automated EBSD with other microstructural probes such as TEM and AFM provides data of greater value than any single technique in isolation. The coupled techniques have been applied to aid in understanding the underlying mechanisms of grain boundary network engineering and the corrosion properties of individual boundaries.

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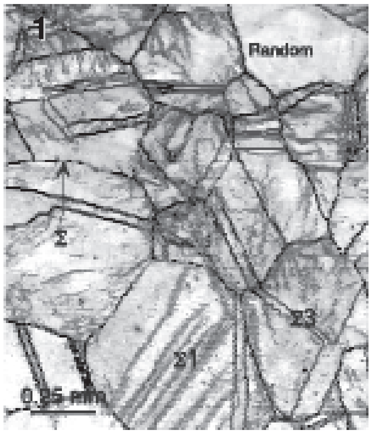

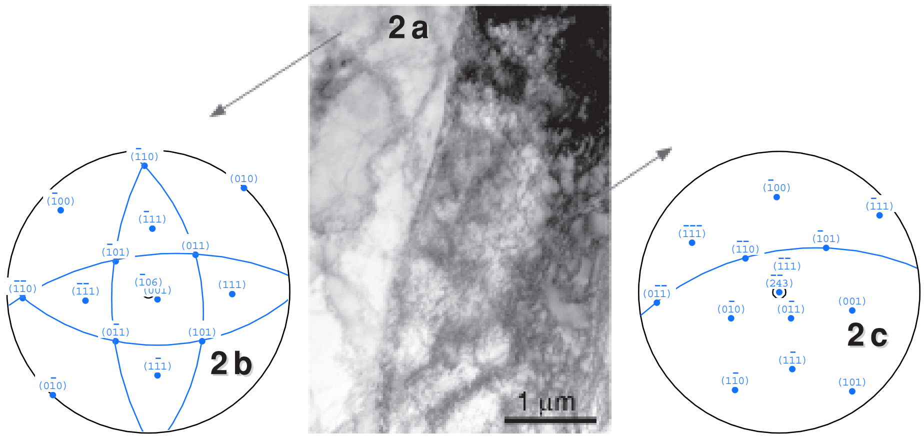

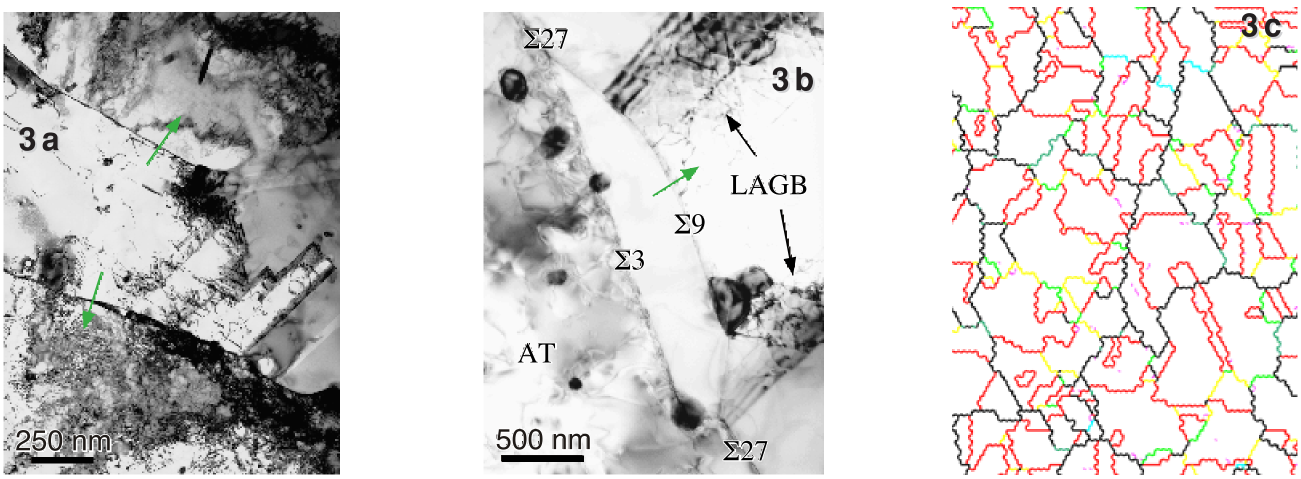

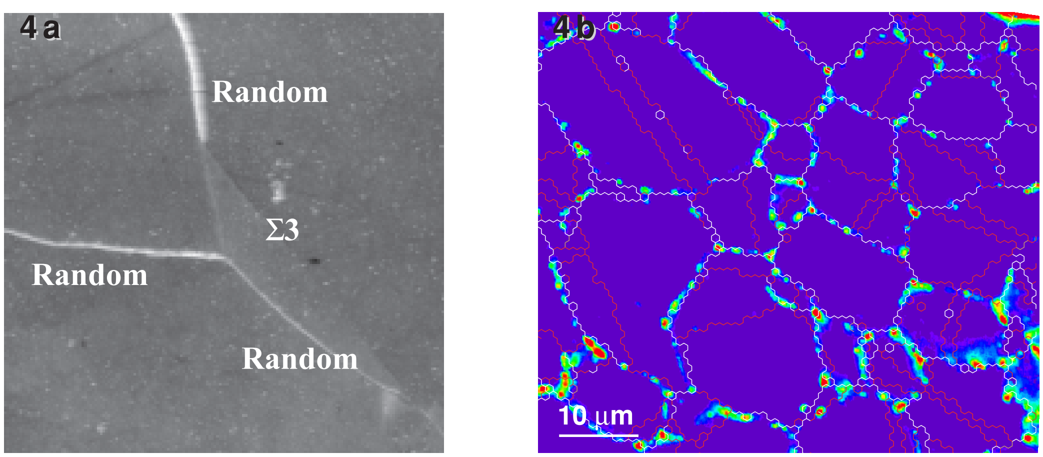

Figure 1. Automated EBSD image of Inconel 600 solution treated and compressed 15%.

Figure 2. (a) TEM image of a grain boundary for the material in Figure 1, (b and c) stereographic projections indicating the orientation of each grain.

Figure 3. (a) Annealing twins in partially recrystallized Inc600, (b) Σ3, Σ9, andΣ27 triple junction, and (c) EBSD image revealing a high fraction of 3-special-boundary triple junctions.

Figure 4. (a) AFM image of corroded Alloy C-22, (b) overlay of EBSD image and AFM topological image of electropolished Inconel 600.